Name I.C. LEWIS / T. PARRU

Notebook Number 195 · 129

Subject Chemistry of Raw Materials, Mixes, Binders + Impregnants

Dates From _____ To _____

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52 **Subject** Initial Data of c/c Composite via BP Process (c/cCOMPBP)
Cross-Reference (if any)

Purpose:

To obtain initial wt and dimension prior to vacuum impregnation w/ a "T-143 TYPE" resin

Mat'l:

c/c composite via BP Process #4-1 : Rec'd from R.S. rocky 4/30/01. From 12th Lawrenceburg Trial. Block 4-1 ~1.5 x 3.0 x 7.0 inches. From 4th block in the series. Made w/ 0.25" long pitch fibers and Reilley 155° pitch, Wood Ratio 85/15 + 5 wt% sulfur based on the pitch weight.

Procedure:

Ultrasonic washed in deionized H_2O 3x, 5min intervals ⇒ Vacuum dried overnight at ~150°C to 2.4mm pressure.

Unloaded & cooled in dessicator. 5/8/01. Weighed & dimensioned 5/11/01 ⇒ loaded into oven at ~150°C, 5.0 SCFH argon purged until loaded in to VI unit.

Weight & Dimension: (5/11/01)

Wt (g)	L_1	L_2	L_3	Ave LEN.	W_1	W_2	W_3	AVE WIDTH	H_1	H_2	H_3	AVE Ht.	VOL. (cc)
1139.84	191.91	192.27	192.02	192.07	83.78	83.36	82.32	83.15	47.21	46.31	46.65	46.86	748.36

⇒ Density = 1.522 g/cc

Comments:

composite contains severe defects, such as fissures and splits. One area appears to contain iron oxide.

VI and Cure: Ref. 19-127-~~55~~ 55d56

Performed and Recorded by: [signature] Date 5/11/01
Directed by: [signature] Date 5/11/01
Read and Understood by: Date

Subject Preparation of ② 900 ml Aliquots of 50/50 by Volume GP-5432/Furfural (clc carrier)
Cross-Reference (if any)

Purpose:
To impregnate 195-129-52 w/a "T-143 Type" resin.

Materials:
GP-5432: Lot # 19588. Rec'd from Georgia-Pacific 9/27/96. LIMS # A96-03635. Stored in Freezer. Ave % Mod MCC (3) = 48.6 (σ = 0.37, n = 3), Brkfld Visc (3) = 157.3 cPs at 71.0°F, PDSC Data: Ref. 195-105-49, TGA Yield (900°C) = 47.5 %, Current Visc = 283.5 cPs at 68.7°F
Furfural: Reagent Grade (Fisher). Rec'd 1/15/01. 2nd Aliquot used 60 ml of ② 500 ml rec'd 5/15/01 ⇒ Balance = 940 ml.

Preparation 1st Aliquot: (5/14/01) - 1L Erlenmeyer Flask. 450 ml GP-5432 + 450 ml Furfural

FLASK (etc) + GP-5432 (3) =	996.3	FLASK (etc) + FURFURAL (3) =	1516.0
FLASK (etc) (3) =	455.9	FLASK (etc) (3) =	996.3
450 ml GP-5432 (3) =	540.4 g	450 ml FURFURAL (3) =	519.7 g ⇒ 50.98 % GP-5432 by wt

Comments:
Mag stirred w/o external heat for 15 min after combining ⇒ Transfer to 32 oz glass jar.

Characterization of the 1st Aliquot: (5/15/01)

Brookfield (LVT) RT Viscosity:

Viscosity = 18.3 cPs at 71.8°F Spindle #1, 60 RPM, Factor = 1

Specific Gravity at RT:

S.G. = 1.190 at 71.8°F

Preparation of 2nd Aliquot: (5/15/01) - 1L Erlenmeyer Flask. 450 ml GP-5432, 450 ml Furfural

FLASK (etc) + GP-5432 (3) =	995.0	FLASK (etc) + FURFURAL (3) =	1512.6
FLASK (etc) (3) =	455.9	FLASK (etc) (3) =	995.0
GP-5432 (3) =	539.1 g	FURFURAL (3) =	517.6 g ⇒ 51.02 % GP-5432 by wt

Solutions combined 5/16/01. Label as 195-129-53 1st Use: Ref. 195-129-55

% Mod MCC of Aliquot #1: Ref. 195-129-54

Performed and Recorded by: _____ **Date** / /
Directed by: _____ **Date**
Read and Understood by: _____ **Date**

Subject % Mod MCC of 195-129-53, 1st Aliquot   (c/c COMP31

Cross-Reference (if any)

Material:

195-129-53, 1st Aliquot : 50/50 by volume, 51.0/49.0 by weight 6P-5432/F...
Prep. 5/14/01, Visc(21) = 18.3 cPs at 71.8°F, SG(21) = 1.190 at 71

% mod MCC Determinations : (5/15/01) - ~1.5g sample size. Cured 1hr at 144°C, 5.0 SCFH

1) CRUC+CHIPS+SAMPLE (21) = 19.1507 CRUC+CHIPS+SAMPLE (144) = 18.3997g ⇒ Yield(144) = 50.5…
CRUC+CHIPS (21) = 17.6308 CRUC+CHIPS+SAMPLE (MCC) = 18.1502g ⇒ Yield(MCC) = 67.5…
SAMPLE (21) = 1.5199g

TOTAL YIELD = (0.5059 × 0.6755) × 100 = 34.17%

2) CRUC+CHIPS+SAMPLE (21) = 18.9635 CRUC+CHIPS+SAMPLE (144) = 18.2000g ⇒ Yield(144) = 49.20
CRUC+CHIPS (21) = 17.4606 CRUC+CHIPS+SAMPLE (MCC) = 17.9770g ⇒ Yield(MCC) = 69.84…
SAMPLE (21) = 1.5029g

TOTAL YIELD = (0.4920 × 0.6984) × 100 = 34.36%

3) CRUC+CHIPS+SAMPLE (21) = 18.3929 CRUC+CHIPS+SAMPLE (MCC) = 17.6460g ⇒ Yield(144) = 50.5
CRUC+CHIPS (21) = 16.8837 CRUC+CHIPS+SAMPLE (MCC) = 17.4073g ⇒ Yield(MCC) = 68.6…
SAMPLE (21) = 1.5092g

TOTAL YIELD = (0.5051 × 0.6869) × 100 = 34.69%

Comments:

All samples were hard at temperature after cure at ~144°C.

Ave Yield(144) = 50.1%, σ = 0.78, n = 3

Ave Yield(MCC) = 68.7%, σ = 1.15, n = 3

Ave Mod MCC = 34.4%, σ = 0.26, n = 3

Performed and Recorded by: [signature] Date

Directed by: [signature] Date

Read and Understood by: Date

Subject VI + Cure to ~200°C of 195-129-52 #4-1 w/ 195-129-53

Cross-Reference (if any)

Purpose:

To density c/c composite w/a phenolic resin/furfural blend. To verify max vol% pickup w/ pitches in the PSC.

Materials:

1) c/c composite: 195-129-52 #4-1 (c/c composite via BP process. From 4th Block in the 12" Lawrenceburg Trial. 0.25" long pitch fibers + Reilley 105 Pitch. Load ratio 85/15 + 5 wt% sulfur based on the pitch wt.) $Wt_{(c)}$ = 1138.84 g, $Vol_{(c)}$ = 748.383 cc, $Dens_{(c)}$ = 1.522 g/cc

2) Impregnant: 195-129-53 (50/50 by volume GP-543X + furfural, Prep. 5/14 & 15/01.

$Vis_{(i)}$ = (17.4) cPs at 74.1°F, $S.G._{(i)}$ = (1.188) at 74.5°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 + 16

Pump-down Data: (5/14-16/01)

DATE	TIME	AREA (mTorr)	Comments
5/14	13:30	15	Load c/c composite from oven (i.e. ~150°C, Atm. Pressure).
"	13:40	4	
"	16:05	16	
5/15	7:20	14	
"	16:00	16	
5/16	7:25	13	
"	8:05	15	charge traps w/ dryice-acetone
"	8:50	10	LDR
"	11:45	10	Begin VI

Impregnation Data: (5/16 & 17/01) - LDR w/ traps charged

LDR: Initial = 10 millitorr
5 min = 21 "
10 min = 27 "
15 min = 34 "

$Vis_{(i)}$ = (17.4) cPs at 74.5°F
$SG_{(i)}$ = (1.188) at 74.5°F

Drop Time = 11:45 (10 mTorr)
Unload Time = 8:45 (5/17/01)
Held at atmospheric pressure for ~21 hrs.

Comments:

570 ml of impregnant in the 500 ml cylindrical funnel.

cont'd next page

Performed and Recorded by: _____ **Date** _____

Directed by: J (Lew **Date** _____

Read and Understood by: _____ **Date** _____

56 **Subject** VI and Cure (~350°C) Data of 195-129-52 #4-1 w/ 195-129-53 (c/c comp #1

Cross-Reference (if any)

Post Impregnation Data: (5/17/01)

Wt(PVI-1) = 1320.11g ⇒ wt Pickup = 181.27g ⇒ wt/o Pickup = 15.92, Vol/o Pickup = 20.40

Al Pan + Screen (E) = 181.4g

Curing Data: (5/17/01) - Cure on small Al Pan w/an s.s. screen to determine amount of run-out.

	TIME	OVEN SET	OVEN TEMP	Comment
*	8:45	48	158	Load into oven. Purge w/argon at 5.0 SCFH (A12)
	9:15	"	156	Wet resin on all visible surfaces. Condensation on oven door.
	9.30	"	154	Impregnant boiling on surfaces, Run-out on screen & in the pan.
	10:05	"	158	Boiling has ceased. Resin is likely cured.
-(2)	10:40	"	156	Unload to desiccator. Set oven at "82°C (180°F). Cool o/c composite ⇒ weigh.
	-	-	-	Wt = 1232.35g ⇒ wt Pickup = 93.5g ⇒ wt/o Pickup = 8.21 (Yd = 51.6%).
	-	-	-	Pan + Screen + Run-out = 188.4g ⇒ Cured Run-out = 7.0g
*	11:55	82	245	Load over (180° Rotation # to bottom).
-(2)	13:55	"	248	Power off. Allow oven to cool to ~150°C ⇒ unload to desiccator
	15:25	OFF	156	Unload to desiccator. Cool overnight ⇒ weigh following morning

Post Curing Data: (5/18/01)

Pan + Screen + Cured Run-out (E) = 188.3g ⇒ Cured Run-out = 6.9g

Wt(E) = 1212.74g ⇒ wt Pickup = 73.9g ⇒ wt/o Pickup = 6.49 (Yd = 40.8%)

⇒ Impregnant Yield (including the run-out) = 44.6% ⇒ Density(E) ≅ 1.620 g/cc

Label 195-129-56, Give to P. Sinocky 5/21/01

Rebake Data: Ref. 195-129-76

Performed and Recorded by: [signature] **Date** 5/21/01

Directed by: [signature] **Date**

Read and Understood by: **Date**

76 **Subject** Rebake Data (10°C/hr to 900°C, 2 hr Hold) of 195-129-56 (BP c/c Comp
Cross-Reference (if any)

Material:

195-129-56: c/c composite via BP process from the 4th Block in the 1st Lawrenceburg trial. 0.25" long pitch fiber + Reilley 15S Pitch. Load Ration 85/15 +5 wt/o solfur based on pitch wt. Initial Data: Ref. 195-129-52 #4-1, VI & Cure Data: Ref. 195-129-55 & 56. Recv'd from P. Sirocky 6/20/01.

Rebake Cycle (per P. Sirocky):

10°C/hr to 900°C, 2hr hold. Block was warm when received. Cooled in desiccator ⇒ weighed & dimensioned.

Rebake Data: (6/20/01)

wt (gm)	L_1	L_2	L_3	AVE. LEN.	W_1	W_2	W_3	AVE WIDTH	H_1	H_2	H_3	AVE Ht.	Vol (cc)
1179.43	192.10	192.32	192.37	192.26	83.98	83.62	82.86	83.49	46.55	46.62	46.60	46.59	742.836

Rebake Density = 1.577 g/cc ⇒ Δ = 0.055 g/cc over "green" density.
Wt/o (green thru bake) = 3.56 %
Vol/o (green thru rebake) = −0.07 % (essentially no change)
Dn/o (green thru rebake) = 3.61 %
Wt/o (cure thru rebake) = −2.75 %

Returned to P. Sirocky 6/20/01. Data communicated via e-mail to D. Hoang, P. Sirocky, & E. Pancost.

Comments:

* Impregnant % wt Yield from VF thru Rebake = (40.59/181.27) × 100 = 22.4 %

Note:

% Mod MCC = 34.4 (σ = 0.26, n = 3) for the impregnant ⇒ The difference is due to run-out and/or weight loss from the pitch binder because it hadn't been to ~900°C yet.

Performed and Recorded by: _____ **Date** / /
Directed by: _____ **Date**
Read and Understood by: _____ **Date**

86 **Subject** Initial data of c/c composites via BP Process (3rd Trial) (c/c Comp.
Cross-Reference (if any)

Purpose:

To obtain initial weights and dimensions prior to vacuum impregnation with silicon (IV) oxide colloidal dispersion for in-situ conversion to SiC.

Materials:

c/c Composite via BP process. Rec'd from P. Sinocky 7/16/01. Two bricks sectioned in ~ half. from the 3rd Lawrenceburg trial. Made w/ 0.25" long K-223SE pitch fibers and Reilley 155 pitch. Load Ration 75/25 w/o solvent. !

Procedure:

Ultrasonic washed in deionized H_2O 3x, 5 min intervals. ⇒ Vacuum dried on at ~166 °C, 0.1 mm pressure.

Air cooled, will not fit in desiccator, ⇒ weighed & dimensioned.

Initial Data: (7/19/01)

File Path = c:\Program Files\Excel\BP c-c Composites\Initial.XLS Sheet = BP III

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-III-1 and BP-III-2. Rec'vd 7/16/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/18/01. Dimensions were obtained with a Starrett No. 123-12 vernier caliper. Hot vacuum dried at 166 °C to 0.1 mm pressure from 7/18 to 7/19/01. Weights obtained on Mettler PN 2210 balance on 7/19/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Den. (g/cc)
3-1-A	916.19	8.173	8.155	8.110	8.146	3.261	3.305	3.304	3.290	1.333	1.302	1.288	1.308	574.297	1.595
3-1-B	823.65	8.196	8.200	8.197	8.198	3.296	3.271	3.248	3.272	1.253	1.273	1.259	1.262	554.502	1.485
AVERAGE =															1.540
STND. DEV. =															0.0777

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Den. (g/cc)
3-2-A	867.13	8.883	8.863	8.837	8.861	3.226	3.265	3.298	3.263	1.183	1.170	1.169	1.174	556.247	1.559
3-2-B	861.58	8.913	8.905	8.896	8.905	3.263	3.281	3.292	3.279	1.196	1.207	1.180	1.194	571.400	1.508
AVERAGE =															1.533
STND. DEV. =															0.0361

Dimensioned: 07/19/01
Hot Vac. Dried: 07/18-19/01
Weighed: 07/19/01
N.B. Ref. No. : 195-129-86

#	Impregnant					NB Reference
3-1-A	Silicon (IV) Oxide Colloidal Dispersion					195-129-88 & 89
3-1-B	"	"	"	"		195-129-92 & 93
3-2-A	"	"	"	"	2VJs	195-129-94 & 95
3-2-B	"	"	"	"	"	195-129-96 & 97

Performed and Recorded by: Date / /
Directed by: Date
Read and Understood by: Date

88 **Subject** VI of 195-129-86 #3-1-A w/ Silicon (III) Oxide Colloidal Dispersion C/C Co

Cross-Reference (if any)

Purpose:

To investigate insitu conversion of carbon to SiC in a c/c composite

Materials:

1) c/c Composite: 195-129-86 #3-1-A: (c/c composite via BP process from the 1st Blo of the 3rd Lawrenceburg trial. 0.25" long K-223SE pitch Fibers and Reilley 15% p. Load Ratio 75/25. No sulfur. Wt(I) = 916.19g, Vol(I) = 574.297cc, Den(I) = 1.595 g

2) Impregnant: Silicon (III) Oxide, 30% in H_2O, colloidal dispersion. (Alfa-Aesar), Lot # A04K09, 0.01 um particles, in liquid, S.A. = 320 m^2/g. Density= Vis(I) = 7.7 cps at 82.1°F, S.G.(I) = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 & 16 Use teflon support and Pyrex pan for drying.

Pump down Data: (7/20-23/01)

DATE	TIME	PRESS (mTorr)	Comments
7/20	12:45	18	Load c/c composite from cold, dry oven.
"	13:00	"	Begin pump-down.
"	14:00	44	
"	15:55	28	
7/23	7:20	21	
"	8:00	"	Charge traps w/ dry ice-acetone.
"	9:05	14	LDR
"	11:50	13	Begin VI

Impregnation Data: (7/23 & 24/01) - LDR w/ traps charged

LDR: Initial = 14 mTorr
5min = 27 "
10min = 38 "
15min = 49 "

Vis(I) = 7.7 cps at 82.1°F
S.G.(I) = 1.216 at 82.1°F

Drop Time = 11:50 (03 m
Unload Time = 8:00 (7/24
Held at atmospheric pres
for ~ 20 1/4 hrs.

Comments:

500 cc cylindrical funnel is full. Return impregnant to ② 1qt. poly-bot

Post Impregnation Data: (7/24/01)

Wt(PVI-1) = 1029.86g ⇒ Wt Pickup = 113.67g ⇒ wt% Pickup = 12.41 Vol% Pickup = 16.

Performed and Recorded by: Date 7

Directed by: J Lewis Date

Read and Understood by: Date

Subject VI of 195-129-86 #3-1-A w/Silicon Diboride Colloidal Dispersion (c/c comp AP) 89

Cross-Reference (if any)

Drying Data: (7/24&25/01) - Oven set "29" (50%). Argon purge 5.0 scfh (A+R)

	TIME	OVEN TEMP°	PRESS (mm)	Comments
*	8:10	108	Atm	Load oven; # to top. Oven set "29" (50%). Purge w/argon at 5.0 scfh (A+R)
(3)	11:10	106	"	Unload oven. Set oven at "38" (50%). Weigh brick hot Co. doesn't fit in dessicator
	—	—	—	Wt = 989.00g ⇒ Wt. Pickup = 72.81g ⇒ Wt% Pickup = 7.95 (Yd = 64.1%)
*	11:30	125	Atm	Load oven; # to bottom. Oven set at "38" (50%). Purge w/argon at 5.0 scfh (A+R)
(2)	13:30	130	"	Unload oven. Set oven at "46" (50%) Weigh brick hot. Install new gasketing.
	—	—	—	Wt = 954.41g ⇒ Wt. Pickup = 38.22g ⇒ Wt% Pickup = 4.17 (Yd = 33.6%)
*	14:20	126 (7)	736.6	Load oven; # to top. Oven set "46" (50%). Vac. pump on. Argon purge off. Reduce pressure.
(07)	7:20	175	0.4	Vac. pump off. Pressurize w/argon. Set oven at "29" (50%). Leave door open to cool.
	—	—	—	Wt = 952.26g ⇒ Wt. Pickup = 36.07g ⇒ Wt% Pickup = 3.94 (Yd = 31.7%)

Comments:

1) After 3 hrs, ~107°C, atm pressure;
No evidence of run-out. Set oven at "38" (50%). Rotate brick 180° (# to bottom).

2) After 3 hrs, ~129°C, atm pressure;
No evidence of run-out. Set oven at "46" (50%). Rotate brick 180° (# to top). Remove old door gasketing and replace w/new gasketing

3) After 17 hrs, ~175°C, vacuum;
Note: by 15:30 (7/24/01) the oven temperature was ~160°C and the pressure was 0.9 mm ⇒ sample dried quickly.
No evidence of run-out. Set oven to "29" (50%) and leave door open to cool.

Comments:

The percent weight yield of the impregnant, in situ, agrees w/that of the solution in crucibles. Ref. 195-129-90.

Label 195-129-89

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Performed and Recorded by: _____ **Date** — / /

Directed by: _____ **Date**

Read and Understood by: _____ **Date**

90 **Subject** Drying of ~10g Samples of Silicon (IV) Oxide, 30% in H_2O, colloidal dispersion (c/c comp
Cross-Reference (if any)

Purpose:

To determine the percent yield by weight of solution to compare w/ in-situ percent weight yield of VIEL c/c composites.

Material:

silicon (IV) oxide, 30% in H_2O, colloidal dispersion (Alfa-Aesar), Lot# A04K09, 0.01 um particles, in liquid. SA = 320 m^2/g. Density = 1.20.
Vis (I) = 7.7 cps at 82.1°F, Spec. Grav (I) = 1.216 at 82.1°F.

Apparatus:

(2) 100 ml porcelain. Al foil covers.

Procedure:

Weigh ~10g into each crucible, containing S.C boiling chips. Cover w/ Al foil. Punch holes in Al foil and obtain total weight. Subtract sample weight to obtain TARE. Processed w/ 195-129-89

Initial Data: (7/24/01)

1) CRUC+CHIPS+SAMPLE (I) = 59.4975 (g) CRUC (etc) + Al Foil (I) = 60.1600
CRUC+CHIPS (I) = 49.3229 CRUC (etc) (I) = 59.4975
SAMPLE (I) = 10.1746g Al Foil (I) = 0.6625g ⇒ TARE = 49.4854

2) CRUC+CHIPS+SAMPLE (I) = 54.7967 (g) CRUC (etc) + Al Foil (I) = 55.4580
CRUC+CHIPS (I) = 44.7620 CRUC (etc) (I) = 54.7967
SAMPLE (I) = 10.0347g Al Foil (I) = 0.6613g ⇒ TARE = 45.4233

Drying Data: (7/24-25/01) - Processed w/ 195-129-89

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:10	108	Atm	Load in oven; in front of Pyrex tray w/ 195-129-89. Purge w/ argon at 5.0 SCFH (air)
-(3)	11:10	106	"	Unload, Cool in desiccator ⇒ weigh. Set oven at "38" (50%).
	—	-	-	(#1) Cruc (etc) Wt = 54.7662g ⇒ Wt = 4.7808g ⇒ Wt Yield = 46.99%
	-	-	—	(#2) Cruc (etc) Wt = 49.9430g ⇒ Wt = 4.5197g ⇒ Wt Yield = 45.04%
*	11:30	125	Atm	Reload; switch sides. Oven set "38" (50%). Purge w/ argon at 5.0 SCFH (AIR)
-(2)	13:30	130	"	Unload. Cool in desiccator ⇒ weigh. Set oven at "46" (50%).
	—	—	-	(#1) Cruc (etc) Wt = 53.2721g ⇒ Wt = 3.2867g ⇒ Wt Yield = 32.30%
	—	—	—	(#2) Cruc (etc) Wt = 48.6710g ⇒ Wt = 3.2477g ⇒ Wt Yield = 32.36%

cont'd next page

Performed and Recorded by: Date
Directed by: Date
Read and Understood by: Date

Subject Drying of ~10g samples of Silicon (IV) Oxide, 30% in H_2O, colloidal dispersion (clc cont. ABP)
Cross-Reference (if any)

Drying Data (cont'd): (7/24+25/01)

	Time	OVEN TEMP	PRESS (mm)	Comments
*	14:20	126 (N)	736.6	Reload; reverse sides. Oven set @ 40°C (1/2). Vac. pump on. Argon purge off.
(07)	7:30	175	0.4	Vac. pump off. Pressurize w/ argon. Unload, cool in desiccator. ⇒ weigh.
	–	–	–	(1) Crucible) Wt = 53.2182g ⇒ Wt = 3.2328g ⇒ Wt Yield = 31.77%
	–	–	–	(2) Crucible) Wt = 48.6127g ⇒ Wt = 3.1894g ⇒ Wt Yield = 31.78%

Ave % Wt Yield = 31.8, σ = 0.01, n = 2

Comments:

Almost exactly the same value for each sample. Same percent weight yield as the in situ % wt. yield for 195-127-89.

Performed and Recorded by: Date
Directed by: Date
Read and Understood by: Date

92 **Subject** VI of 195-129-86 #3-1-B w/ Silicon (III) oxide, Colloidal Dispersion (c/c Co

Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

1) c/c Composite: 195-129-86 #3-1-B; C/C composite via BP process from the 1st Block of the 3rd Lawrenceburg trial. 0.25" long K-223-SE pitch fiber and Reilley 155, Load ratio 75/25. No sulfur. Wt(c) = 823.65g, Vol(c) = 554.502 cc, Density = 1.485 g/

2) Impregnant: Silicon (III) oxide, 30% in H_2O, colloidal dispersion. (Alfa-Aesar), Lot# A04K09 0.01 um particles, in liquid. SA = 320 m^2/g. Density = 1.20. Previous use: 195-129-8 Vis(c) = 7.7 cps at 82.1 °F, S.G.(c) = 1.216 at 82.1 °F.

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-129-88

Pump-down Data:

DATE	TIME	PRESS (mTorr)	Comments
7/23	13:15	21	Load block from hot oven (~110°C, Atm)
"	13:25	"	Begin pump-down
"	14:25	45	
7/24	7:10	24	
"	8:25	23	charge traps w/dryice-acetone
"	8:55	15	LDR
"	11:47	13	Begin VI

Impregnation Data: (7/23+24/01) - LDR w/traps charged.

LDR: Initial = 14 mTorr Vis = (7.8 cps at 78.0°F Drop Time = 11:45 (13 mTorr

5min = 27 " S.G. = (1.216) at 78.0°F Unload Time = 7:45 (7/25/01

10min = 38 " Held at atmospheric pressure f

15min = 49 " ~20 hrs.

Comments:

500 ml cylindrical funnel Filled (ie. ~675 ml)

Post-Impregnation Data: (7/25/01)

$Wt_{(PVI-1)}$ = 950.80g ⇒ Wt Pickup = 127.15g ⇒ Wt% Pickup = (15.44) Vol% Pickup = 18.86

Performed and Recorded by: Date

Directed by: Date

Read and Understood by: Date

Subject VI at 195-129-86 #3-1-B w/silicon (III) oxide, Colloidal Dispersion (c/c comp BP)
Cross-Reference (if any)

Drying Data: (7/25+26/01) - Oven set "29" (50%). Purge w/argon at 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:00	107	Atm	Load oven. # to top. Oven set "29" (50%). Argon purge 5.0 SCFH (AIR)
(3)	11:00	108	"	Unload oven. Oven set "38" (50%). Weigh block hot.
	—	—	—	Wt = 907.62g ⇒ Wt. Pickup = 83.97g ⇒ Wt% Pickup = 10.19 (Yd = 66.0%)
*	11:20	129	Atm	Load oven; # to bottom. Oven set "38" (50%). Argon purge 5.0 SCFH (AIR)
(2)	13:20	132	"	Unload oven. Oven set "46" (50%). Weigh block hot. Install new gasketing.
	—	—	—	Wt = 871.76g ⇒ Wt Pickup = 48.11g ⇒ Wt% Pickup = 5.84 (Yd = 37.8%)
*	13:40	136(A)	736.9	Load oven; # to top. Oven set "46" (50%). Vac. pump on. Argon purge off.
(18)	7:40	168	0.4	Vac. pump off. Pressurize w/argon. Set oven at "29" (50%).
	—	—	—	Wt = 863.89g ⇒ Wt. Pickup = 40.24g ⇒ Wt% Pickup = 4.89 (Yd = 31.6%)

comments:

1) After 3 hrs, ~108°C, atm. pressure;
Rotate brick 180° (# to bottom). Set oven at "38" (50%). No evidence of run-out
Similar % wt Yield to 195-129-89 (i.e. 64.1%).

2) After 2 hrs, ~131°C, atm pressure;
Scrape off old gasketing and install new, set oven to "46" (50%). Rotate brick 180° (# to top). No evidence of run-out. H_2O condensed on oven door; wiped dry.

3) After 18 hrs, ~168°C, vacuum;
No evidence of run-out. Oven set to "29" (50%). Leave door open to cool.

Label 195-129-93
Graph Data: Ref. 195-130-63

Performed and Recorded by: Date / /
Directed by: Date
Read and Understood by: Date

94 **Subject:** 1st VI of 195-129-86 #3-2-A w/ silicon (III) oxide, colloidal dispersion (c/c com

Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

1) c/c Composite 195-125-86 #3-2-A (c/c composite via BP process. From block #2 of the 3rd Lawrenceburg trial. 0.25" long K-223SE pitch Fibers. Reilley 155 pitch. Load Ratio = 75/25 w/o sulfur. Wt(1) = 867.13g, Vol(1) = 556.247 cc, Dens(1) = 1.557 g/cc

2) Impregnant: Silicon (III) oxide, 20% in H_2O, colloidal Dispersion. (Alfa-Aesar), Lot # A04Y 0.01 um particles, in liquid. SA = 320 m^2/g. Density = 1.20, Previous use: 7/25/01. Visc(2) = 7.7 cps at 82.1°F, SG(2) = 1.216 at 82.1°F.

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 + 16 * Processed w/ 195-129-86 #3-2-B

Pump down Data: (7/24-25/01)

DATE	TIME	PRESS (mm)	Comments
7/24	13:20	19	Load from oper lab
"	13:35	"	Begin pump-down
"	14:35	340	"High" pressure ⇒ brick picked up moisture in lab
"	15:55	~205	
7/25	7:20	37	
"	8:35	33(4)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:45	18	Begin VI

Impregnation Data: (7/25+26/01) - LDR cold traps charged

LDR: Initial = 22 mTorr
5 min = 46 "
10 min = 60 "
15 min = 74 "

Visc(2) = 7.7 cps at 78.0°F
S.G.(2) = 1.220 at 78.0°F

Drop Time = 11:45 (16 mTo
Unload Time = 8:15 (7/26/0
He(g) at atmospheric pressu
for ~ 20 1/2 hrs.

Comments:

Filled second cylindrical funnel.

Post-Impregnation Data: (7/26/01)

Wt (PVI-1) = 987.35g ⇒ Wt Pickup = 120.22g ⇒ Wt% Pickup = 13.86, Vol% Pickup = 17.

Performed and Recorded by: _____ **Date**

Directed by: _____ **Date**

Read and Understood by: _____ **Date**

Subject 2nd VI of 195-129-86 #3-2-A w/ 5.1 cm colloidal dispersion (ck. comp #)
Cross-Reference (if any)

Drying Data: (7/26+27/01) - Oven set "29" (50%). Purge w/argon at 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:30	106	Atm	Load oven; # to top. Oven set "29" (50%). Argon purge at 5.0 SCFH (AIR)
-(1)	11:30	120	"	Unload oven. Oven set "38" (50%). Weigh block hot.
	-	-	-	Wt = 944.48 g ⇒ wt Pickup = 77.35 g ⇒ wt/o Pickup = 8.92 (Yd = 64.3%).
*	11:45	150	Atm	Load oven; # to bottom, front of tray. Oven set "38" (50%). Argon purge 5.0 SCFH
-(2)	13:45	138	"	Unload oven. Oven set "46" (50%). Weigh block hot. Install new gasketing.
	-	-	-	Wt = 906.75 g ⇒ wt Pickup = 39.62 g ⇒ wt/o Pickup = 4.57 (Yd = 33.0%).
*	14:30	176	742.0	Load oven; # to top, back of tray. Vac. pump on. Argon purge off. Reduce pressure.
-(3)	8:30	166	0.3	Vac. pump off. Pressurize w/argon. Set oven at "29" (50%).
	-	-	-	Wt = 905.02 g ⇒ wt Pickup = 37.89 g ⇒ wt/o Pickup = 4.37 (Yd = 31.5%)

Comments:

1) After 3 hrs, ~113 °C, atm. pressure;
No evidence of run-out. Set oven at "38" (50%). Weigh block hot. Rotate 180°; # to bottom. Reverse position w/ 195-129-86 #3-2-B (i.e. front of Pyrex tray).

2) After 2 hrs, ~147 °C, atm pressure;
No evidence of run-out. Set oven at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position w/ "B" (i.e. rear of Pyrex tray). Remove old gasketing and install new.

3) After 18 hrs, ~171 °C, vacuum;
Set oven at "29" (50%). Weigh block hot ⇒ reload w/ "B" comparison into VI unit for 2nd impregnation

Comments:
% yield of the impregnant agrees w/ previous (2) blocks; 31.7%, 31.6%, and 31.5%.

Label 195-129-95 #3-2-A 2nd VI: Ref. 195-129-99+100

Performed and Recorded by: L. [signature] Date / /
Directed by: J Clair Date
Read and Understood by: Date

Subject VI of 195-129-86 #3-2-B w/ Silicon (IV) oxide, colloidal dispersion (c/c comp.)
Cross-Reference (if any)

Purpose:
Ref. 195-129-88

Materials:
1) c/c Composite 195-129-86 #3-2-B (c/c composite via BP process. From block #2 of the 3rd Lawrenceburg trial, 0.25" long K-223 SE pitch fibers. Railley 15V pitch. Load ratio = 75/25 w/o sulfur. Wt(2) - 861.58g, Vol(2) - 571.400cc, Den(2) = 1.508 g/cc
2) Impregnant: Silicon (IV) oxide, 30% in H_2O, colloidal dispersion. (Alfa-Aesar), Lot # A01 0.01um particles, in liquid. SA = 320 m²/g. Density = 1.20, Previous use: 7/26/01. Visc(2) - 7.7 cps at 82.1°F, S.G.(2) - 1.216 at 82.1°F

Apparatus:
Ref. 195-128-15
Procedure:
Ref. 195-120-15 & 16 *Processed w/ 195-129-86 # 3-2-A

Pump-down Data: (7/24 & 25/01)

DATE	TIME	PRESS (mTorr)	Comments
7/24	13:20	19	Load from open lab w/ #3-2-A
"	13:35	"	Begin pump-down.
"	14:35	~390	"High" pressure ⇒ blocks picked up moisture in lab.
"	15:55	~200	
7/25	7:20	37(L)	
"	8:35	33(L)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:45	18	Begin VI

Impregnation Data: (7/25 & 26/01) - LDR w/ traps charged
LDR: Initial = 22 mTorr
5 min = 46 "
10 min = 60 "
15 min = 74 "

Visc(2) 7.4 cps at 78.0°F
SG(2) = (1.220) at 78.0°F

Drop Time = 11:45 (18 mTorr)
Unload Time = 8:15 (7/26/01)

Comments:
Filled 500 ml cylindrical funnel (i.e. ~675 ml).

Post Impregnation Data: (7/26/01)

Wt (PVI-1) = 995.39g ⇒ Wt Pickup = 133.81g ⇒ wt% Pickup (15.53), Vol% Pickup (19.20)

Performed and Recorded by: [signature] Date:
Directed by: [signature] Date
Read and Understood by: Date

Subject VI of 195-129-86 #3-2-B w/5% iron(III) oxide, colloidal dispersion (clc comp BP)

Cross-Reference (if any)

Phys. Data: (7/26-27/01) - Oven set "29" (50%). Argon purge 5.0 scfh (AIR). Covers w/195-129-95

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:30	106	Atm	Load oven; # to top. Oven set "29" (50%), Argon purge at 5.0 scfh (AIR).
(3)	11:30	120	"	Unload oven. Set oven "38" (50%). Weigh block hot.
	—	—	—	Wt = 950.91g ⇒ wt Pickup = 89.33g ⇒ wt/o Pickup = 10.37 (Yld = 66.8%)
*	11:45	150	Atm	Load oven; # to bottom, back of tray. Oven set "38" (50%). Argon purge 5.0 scfh.
(2)	13:45	138	"	Unload oven. Set oven "46" (50%). Weigh block hot. Install new gasketing.
	—	—	—	Wt = 906.48g ⇒ wt Pickup = 44.90g ⇒ wt/o Pickup = 5.21 (Yld = 33.6%)
*	14:30	176	742.0	Load oven; # to top, front of tray. Oven set "46" (50%). Vac. pump on. Argon purge off.
(18)	8:30	166	0.3	Vac. pump off. Pressurize w/argon. Set oven "29" (50%).
	—	—	—	Wt = 903.59g ⇒ wt. Pickup = 42.01g ⇒ wt/o Pickup = 4.88 (Yld = 31.4%)

Comments:

1) After 3 hrs, ~113°C, atm pressure;
No evidence of run-out. Set oven at "38" (50%). Weigh block hot. Rotate 180°; # to bottom. Reverse position in Pyrex tray w/195-129-86 #3-2-A. (ie. back of tray).

2) After 2 hrs, ~144°C, atm pressure;
No evidence of run-out. Set oven at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position in Pyrex tray (ie. front of tray). Renew old door gasketing and install new.

3) After 18 hrs, ~171°C, vacuum;
Set oven "29" (50%). Weigh block hot ⇒ reload w/195-129-95 #3-2-A into VI unit for 2nd impregnation.

Comments:
% yield of impregnant agrees w/previous ③ blocks; 31.7%, 31.6%, and 31.5%.

Label 195-129-97 #3-2-B 2nd VI: Ref. 195-130-01202

Performed and Recorded by: _____ **Date** / /

Directed by: _____ **Date** / /

Read and Understood by: _____ **Date** / /

98 **Subject** Initial Data of Graphitized c/c Composites via BP Process (4th Trial) GK

Cross-Reference (if any)

Purpose:

To obtain the initial weights and dimensions prior to vacuum impregnation with "T-143" type phenolic/furfural resin blend for densification.

Materials:

c/c Composites via BP process. Rec'd from P. Stracky 7/25/01. Two sections, both graphitized. Section "A-1" had one pitch impregnation, section "B-1" did not have a PI. Both graphitized to ~3000°C

Made w/ K-2235E 0.25" long fibers and Ashland 15S pitch. Load Ratio = 75/2 w/o sulfur. Brick 13 of 4th trial.

Procedure:

Essentially same as 195-128-86. Except cooled in dessicator

Initial Data: (7/27/01)

File Path = C:\Program Files\Excel\BP c-c composites\Initial.XLS Sheet = BP II

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-IV-13 A1 and BP-IV-13 B1. Rec'vd 7/25/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/26/01. Dimensions were obtained with a Mitutoyo Model CD-8"CS digital caliper.
Hot vacuum dried at 124 °C to 0.4 mm pressure from 7/26 to 7/27/01.
Weights obtained on Mettler PN 2210 balance on 7/27/01.
Note: Both samples have been graphitized to ~3000°C. A1 has one PI. B1 has no PI.

Sample I.D.	Weight (g)	L1 (mm)	L2 (mm)	L3 (mm)	Ave. Length (mm)	W1 (mm)	W2 (mm)	W3 (mm)	Ave. Width (mm)	H1 (mm)	H2 (mm)	H3 (mm)	Ave. Height (mm)	Vol. (cc)	Den. (g/cc)
4-13-A1	277.03	108.95	108.89	108.98	108.94	93.90	93.92	93.95	93.92	15.65	16.01	16.08	15.91	162.825	1.701
4-13-B1	255.92	107.20	106.19	105.13	106.17	96.81	97.06	97.50	97.12	16.48	16.15	15.66	16.10	165.987	1.542

Dimensioned: 07/27/01
Hot Vac. Dried: 07/26-27/01
Weighed: 07/27/01
N.B. Ref. No. : 195-129-98

#	Impregnant	NB Ref.
4-13 A1	195-129-53 (w/50 by vol/o GP5432/Furfural)	195-130-03 & 04
4-13-B1	" " " " " / "	195-130-05 & 06

Performed and Recorded by: [signature] **Date**
Directed by: [signature] **Date**
Read and Understood by: **Date**